



Application Note HPR-PE-04

COAL (Partial digestion)

Summary

This method provides the acid digestion of the sample in a closed vessel device using temperature control microwave heating for the metal determination by spectroscopic methods.

Instrumentation

Microwave acid digestion apparatus

Milestone Ethos or Start labstation with internal temperature sensor, 640-260 terminal with easyCONTROL software installed and HPR1000/10S high pressure segmented rotor.

Sample weight

Up to 0.25 g

Reagents

3 ml of HCl 37%, 3 ml of HF 40%,
9 ml of HNO₃ 65%.

Procedure

1. Place a TFM vessel on the balance plate, tare it and weigh of the sample.
2. Introduce the TFM vessel into the HTC safety shield.
3. Add the acids; if part of the sample stays on the inner wall of the TFM vessel, wet it by adding acids drop by drop, then gently swirl the solution to homogenize the sample with the acids.
4. Close the vessel and introduce it into the rotor segment, then tighten by using the torque wrench.
5. Insert the segment into the microwave cavity and connect the temperature sensor.

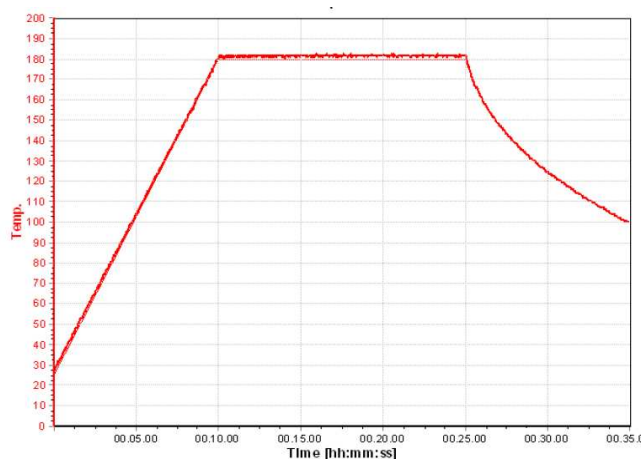
6. Run the microwave program to completion.
7. Cool the rotor by air or by water until the solution reaches room temperature.
8. Open the vessel and transfer the solution to a marked flask.

Microwave program

Step	Time	T1	T2 ⁽¹⁾	P ⁽²⁾	Power
1	00:10:00	180°C	100°C	45 bar	Max power*
2	00:15:00	180°C	100°C	45 bar	Max power*

(1), (2) Optional sensors

Temperature profile



Note

*Max power: 1500W for Ethos and 1200W for Start units.

Use up to 500 Watt for operations with 3 or less vessels simultaneously.

This procedure is only a guideline and it may need to be modified or changed to obtain the required results on your sample.

Milestone suggest to use pressure sensor (P) only for method developments or unknown samples. See the pressure sensor user manual for more information.

Always use hand, eye and body protection when operating with the microwave system.